

## Refine Search

### Search Results -

Terms	Documents
L7 AND (514/\$ OR 560/\$ OR 562/\$)	16

**Database:** US Pre-Grant Publication Full-Text Database  
US Patents Full-Text Database  
US OCR Full-Text Database  
EPO Abstracts Database  
JPO Abstracts Database  
Derwent World Patents Index  
IBM Technical Disclosure Bulletins

**Search:** L8

Refine Search

Recall Text      Clear      Interrupt

### Search History

DATE: Tuesday, February 05, 2008    [Purge Queries](#)    [Printable Copy](#)    [Create Case](#)

<u>Set Name</u>	<u>Query</u>	<u>Hit Count</u>	<u>Set Name</u>
side by side			result set
DB=PGPB,USPT,USOC,EPAB,JPAB,DWPI,TDBD; PLUR=YES; OP=ADJ			
<u>L8</u>	L7 AND (514/\$ OR 560/\$ OR 562/\$)	16	<u>L8</u>
<u>L7</u>	L5 AND PAIN\$9 AND INFLAM\$9	21	<u>L7</u>
<u>L6</u>	L5 AND PAIN AND INFLAM\$9	21	<u>L6</u>
<u>L5</u>	L4 AND URINARY INCONTINENCE	32	<u>L5</u>
<u>L4</u>	L3 AND L2	2255	<u>L4</u>
<u>L3</u>	(N-HYDROXY THIOUREA.TI. OR UREA.TI.) AND AMIDE	2255	<u>L3</u>
<u>L2</u>	(N-HYDROXY THIOUREA OR UREA) AND AMIDE	88358	<u>L2</u>
DB=PGPB; PLUR=YES; OP=ADJ			
<u>L1</u>	20050288369	1	<u>L1</u>

END OF SEARCH HISTORY

## Hit List

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Search Results - Record(s) 1 through 10 of 16 returned.

1. Document ID: US 20070244178 A1

L8: Entry 1 of 16

File: PGPB

Oct 18, 2007

PGPUB-DOCUMENT-NUMBER: 20070244178

PGPUB-FILING-TYPE:

DOCUMENT-IDENTIFIER: US 20070244178 A1

TITLE: Process for the Preparation of Indazolyl Ureas that Inhibit Vanilloid Subtype1 (VR1) Receptors

PUBLICATION-DATE: October 18, 2007

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Lukin; Kirill A.	Vernon Hills	IL	US
Hsu; Margaret Chi-Ping	Lake Forest	IL	US
Fernando; Dilanie P.	Lake Forest	IL	US
Kotecki; Brian J.	Oak Creek	WI	US
Leanna; Marvin R.	Grayslake	IL	US

US-CL-CURRENT: 514/406; 548/361.1

[Full](#) [Title](#) [Citation](#) [Front](#) [Review](#) [Classification](#) [Date](#) [Reference](#) [Sequences](#) [Attachments](#) [Claims](#) [KMC](#) [Drawn D](#)

2. Document ID: US 20070167458 A1

L8: Entry 2 of 16

File: PGPB

Jul 19, 2007

PGPUB-DOCUMENT-NUMBER: 20070167458

PGPUB-FILING-TYPE:

DOCUMENT-IDENTIFIER: US 20070167458 A1

TITLE: Tetrahydro-naphthalene and urea derivatives

PUBLICATION-DATE: July 19, 2007

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Bouchon; Axel	Koln		DE
Diedrichs; Nicole	Velbert		DE
Hermann; Achim	Duseldorf		DE

Lustig; Klemens	Wuppertal	DE
Meier; Heinrich	Wuppertal	DE
Pernerstorfer; Josef	Hilden	DE
Reissmuller; Elke	Wuppertal	DE
De Vry; Jean	Stolberg	DE
Mogi; Muneto	Ibaraki-ken	JP
Urbahns; Klaus	Lund	SE
Yura; Takeshi	Nara-ken	JP
Fujishima; Hiroshi	Nara-ken	JP
Tajimi; Masaomi	Aichi-ken	JP
Yamamoto; Noriyuki	Osaka-fu	JP
Yuasa; Hiroaki	Kyoto	JP
Gupta; Jang	Dusseldorf	DE
Tsukimi; Yasuhiro	Hyogo-ken	JP
Hayashi; Fumihiko	Tokyo	JP

US-CL-CURRENT: 514/255.01; 544/388, 546/205, 546/219

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [KMC](#) | [Drawn D](#)

3. Document ID: US 20070078156 A1

L8: Entry 3 of 16

File: PGPB

Apr 5, 2007

PGPUB-DOCUMENT-NUMBER: 20070078156

PGPUB-FILING-TYPE:

DOCUMENT-IDENTIFIER: US 20070078156 A1

TITLE: Derivatives of n-(1h-indazolyl)-and n-(1h-indolyl)-urea as well as related compounds as modulators of the vanilloid-1 receptor (vr1) for treatment of pain

PUBLICATION-DATE: April 5, 2007

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Fletcher; Stephen Robert	Bishops Stortford		GB
Hollingworth; Gregory John	Brentwood		GB
Jones; Brian A.	Saffron Walden		GB
Moyes; Richard Christopher	Hertford		GB
Rogers; Lauren	Braintree		GB

US-CL-CURRENT: 514/301; 514/302, 514/303, 546/112, 546/114, 546/116, 546/119, 564/1

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [KMC](#) | [Drawn D](#)

4. Document ID: US 20060063775 A1

L8: Entry 4 of 16

File: PGPB

Mar 23, 2006

PGPUB-DOCUMENT-NUMBER: 20060063775  
PGPUB-FILING-TYPE:  
DOCUMENT-IDENTIFIER: US 20060063775 A1

TITLE: Urea derivatives as calcium channel blockers

PUBLICATION-DATE: March 23, 2006

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Pajouhesh; Hassan	Vancouver		CA
Pajouhesh; Hossein	Burnaby		CA
Ding; Yanbing	Vancouver		CA
Snutch; Terrance P.	Vancouver		CA

US-CL-CURRENT: 514/253.12; 514/255.01, 544/360, 544/386

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [KWMC](#) | [Drawn D](#)

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5. Document ID: US 20050288369 A1

L8: Entry 5 of 16

File: PGPB

Dec 29, 2005

PGPUB-DOCUMENT-NUMBER: 20050288369  
PGPUB-FILING-TYPE: new  
DOCUMENT-IDENTIFIER: US 20050288369 A1

TITLE: Novel n-hydroxy thiourea, urea and amide compounds and the pharmaceutical compositions comprising the same

PUBLICATION-DATE: December 29, 2005

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Lee, Jee-Woo	Seoul		KR

US-CL-CURRENT: 514/534; 514/575, 560/41, 562/621

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [KWMC](#) | [Drawn D](#)

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6. Document ID: US 20050187291 A1

L8: Entry 6 of 16

File: PGPB

Aug 25, 2005

PGPUB-DOCUMENT-NUMBER: 20050187291  
PGPUB-FILING-TYPE: new  
DOCUMENT-IDENTIFIER: US 20050187291 A1

TITLE: Aminotetralin-derived urea modulators of vanilloid VR1 receptor

PUBLICATION-DATE: August 25, 2005

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Codd, Ellen	Blue Bell	PA	US
Dax, Scott L.	Landenberg	PA	US
Jetter, Michele	Norristown	PA	US
McDonnell, Mark	Lansdale	PA	US
McNally, James J.	Souderton	PA	US
Youngman, Mark	Warminster	PA	US

US-CL-CURRENT: 514/521; 514/563, 514/595

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7. Document ID: US 20050154230 A1

L8: Entry 7 of 16

File: PGPB

Jul 14, 2005

PGPUB-DOCUMENT-NUMBER: 20050154230

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20050154230 A1

TITLE: Urea derivatives

PUBLICATION-DATE: July 14, 2005

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Yura, Takeshi	Handa-shi		JP
Mogi, Muneto	Nara-shi		JP
Ikegami, Yuka	Kyoto-shi		JP
Masuda, Tsutomu	Handa-shi		JP
Kokubo, Toshio	Nara-shi		JP
Urbahns, Klaus	Lund		SE
Yoshida, Nagahiro	Soraku-gun		JP
Marumo, Makiko	Nara-shi		JP
Shirou, Masahiro	Cambridge		GB
Tajimi, Masaomi	Chita-shi		JP
Takeshita, Keisuke	Takamatsu-shi		JP
Moriwaki, Toshiya	Ikoma-shi		JP
Tsukimi, Yasuhiro	Amagasaki-shi		JP

US-CL-CURRENT: 564/52

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [KWMC](#) | [Drawn D](#)

8. Document ID: US 20050119304 A1

L8: Entry 8 of 16

File: PGPB

Jun 2, 2005

PGPUB-DOCUMENT-NUMBER: 20050119304  
 PGPUB-FILING-TYPE: new  
 DOCUMENT-IDENTIFIER: US 20050119304 A1

TITLE: Urea derivatives

PUBLICATION-DATE: June 2, 2005

## INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Yura, Takeshi	Aichi-ken		JP
Mogi, Muneto	Nara-ken		JP
Ikegami, Yuka	Kyoto-shi		JP
Masuda, Tsutomu	Aichi-ken		JP
Kokubo, Toshio	Nara-ken		JP
Urbahns, Klaus	Lund		SE
Yoshida, Nagahiro	Kyoto-fu		JP
Marumo, Makiko	Nara-ken		JP
Shiroo, Masahiro	Cambridge		GB
Tajimi, Masaomi	Aichi-ken		JP
Takeshita, Keisuke	Kagawa-ken		JP
Moriwaki, Toshiya	Nara-ken		JP
Tsukimi, Yasuhiro	Hyogo-ken		JP

US-CL-CURRENT: 514/314; 514/367, 514/419

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMTC	Draw D
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 9. Document ID: US 20050107388 A1

L8: Entry 9 of 16

File: PGPB

May 19, 2005

PGPUB-DOCUMENT-NUMBER: 20050107388  
 PGPUB-FILING-TYPE: new  
 DOCUMENT-IDENTIFIER: US 20050107388 A1

TITLE: Heteroaromatic urea derivatives as vr-1receptor modulators for treating pain

PUBLICATION-DATE: May 19, 2005

## INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Brown, Rebecca Elizabeth	Cambridge		GB
Doughty, Victoria Alexandra	Stanstead		GB
Hollingworth, Gregory John	Brentwood		GB
Jones, A. Brian	Saffron Walden		GB
Lindon, Matthew John	Stevenage		GB

Moyes, Christopher Richard	Hertford	GB
Rogers, Lauren	Braintree	GB

US-CL-CURRENT: 514/248; 514/210.21, 514/227.8, 514/232.5, 514/249, 514/252.17,  
514/253.06, 514/266.1, 514/310, 514/311, 544/114, 544/126, 544/284, 544/353,  
544/363, 544/60

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10. Document ID: US 20040157865 A1

L8: Entry 10 of 16

File: PGPB

Aug 12, 2004

PGPUB-DOCUMENT-NUMBER: 20040157865

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20040157865 A1

TITLE: Naphthol, quinoline and isoquinoline-derived urea modulators of vanilloid VR1 receptor

PUBLICATION-DATE: August 12, 2004

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Codd, Ellen	Blue Bell	PA	US
Dax, Scott L.	Landenberg	PA	US
Jetter, Michele	Lansdale	PA	US
McDonnell, Mark	Souderton	PA	US
McNally, James J.	Warminster	PA	US
Youngman, Mark			US

US-CL-CURRENT: 514/266.1; 514/310, 514/311, 544/286, 546/153

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Sequences](#) | [Attachments](#) | [Claims](#) | [KMC](#) | [Drawn D](#)

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Terms	Documents
L7 AND (514/\$ OR 560/\$ OR 562/\$)	16

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11. Document ID: US 20030236280 A1

L8: Entry 11 of 16

File: PGPB

Dec 25, 2003

PGPUB-DOCUMENT-NUMBER: 20030236280

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20030236280 A1

TITLE: Aminotetralin-derived urea modulators of vanilloid VR1 receptor

PUBLICATION-DATE: December 25, 2003

#### INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Codd, Ellen	Blue Bell	PA	US
Dax, Scott L.	Landenberg	PA	US
Jetter, Michele	Norristown	PA	US
McDonnell, Mark	Lansdale	PA	US
McNally, James J.	Souderton	PA	US
Youngman, Mark	Warminster	PA	US

US-CL-CURRENT: 514/310; 514/319; 546/143, 546/205

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Drawn D
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12. Document ID: US 7285563 B2

L8: Entry 12 of 16

File: USPT

Oct 23, 2007

US-PAT-NO: 7285563

DOCUMENT-IDENTIFIER: US 7285563 B2

TITLE: Heteroaromatic urea derivatives as VR-1 receptor modulators for treating pain

#### PRIOR-PUBLICATION:

DOC-ID DATE

US 20050107388 A1 May 19, 2005

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Drawn D
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13. Document ID: US 7183411 B2

L8: Entry 13 of 16

File: USPT

Feb 27, 2007

US-PAT-NO: 7183411

DOCUMENT-IDENTIFIER: US 7183411 B2

TITLE: Naphthol, quinoline and isoquinoline-derived urea modulators of vanilloid VR1 receptor

## PRIOR-PUBLICATION:

DOC-ID DATE

US 20040157865 A1 August 12, 2004

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC	Drawn D
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 14. Document ID: US 6984647 B2

L8: Entry 14 of 16

File: USPT

Jan 10, 2006

US-PAT-NO: 6984647

DOCUMENT-IDENTIFIER: US 6984647 B2

TITLE: Aminotetralin-derived urea modulators of vanilloid VR1 receptor

## PRIOR-PUBLICATION:

DOC-ID DATE

US 20030236280 A1 December 25, 2003

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC	Drawn D
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 15. Document ID: US 5661146 A

L8: Entry 15 of 16

File: USPT

Aug 26, 1997

US-PAT-NO: 5661146

DOCUMENT-IDENTIFIER: US 5661146 A

TITLE: Substituted dibenzoxazepine urea compounds, pharmaceutical compositions and methods of use

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC	Drawn D
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 16. Document ID: US 5449675 A

L8: Entry 16 of 16

File: USPT

Sep 12, 1995

US-PAT-NO: 5449675

DOCUMENT-IDENTIFIER: US 5449675 A

TITLE: Substituted dibenzoxazepine and dibenzthiazepine urea compounds, pharmaceutical compositions and methods of use

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Description](#) | [Chemical](#) | [Claims](#) | [KOMC](#) | [Drawn D.](#)

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Terms

Documents

L7 AND (514/\$ OR 560/\$ OR 562/\$)

16

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(FILE 'HOME' ENTERED AT 15:25:46 ON 05 FEB 2008)

FILE 'CAPLUS' ENTERED AT 15:25:53 ON 05 FEB 2008  
L1 STRUCTURE uploaded  
S L1

FILE 'REGISTRY' ENTERED AT 15:26:23 ON 05 FEB 2008  
L2 7979 S L1 FULL

FILE 'CAPLUS' ENTERED AT 15:26:25 ON 05 FEB 2008  
L3 1042 S L2 FULL  
L4 708 S L3 AND PY<2002  
L5 403 S L4 AND (PHENYL? OR BENZYL OR OLEYL)  
L6 1 S L5 AND ( TERT-BUTYLPHENYL OR DIMETHYLBENZYL OR OLEYL)

FILE 'CAPLUS' ENTERED AT 16:37:09 ON 05 FEB 2008  
L7 STRUCTURE uploaded  
S L7

FILE 'REGISTRY' ENTERED AT 16:37:56 ON 05 FEB 2008  
L8 362 S L7 FULL

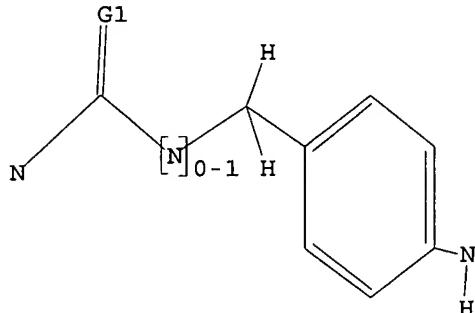
FILE 'CAPLUS' ENTERED AT 16:37:57 ON 05 FEB 2008  
L9 110 S L8 FULL  
L10 54 S L9 AND PY<2002  
L11 1 S L10 AND (BUTYLPHENYL OR DIMETHYLPHENYLPROPYL OR OLEYL)  
L12 0 S L10 AND (BUTYLBENZYL OR DIMETHYLPHENYLPROPYL OR OLEYL)

=>

10/923,271

STRUCTURE UPLOADED

=> d  
L1 HAS NO ANSWERS  
L1 STR



G1 O,S

Structure attributes must be viewed using STN Express query preparation.

=> s l1 full  
REGISTRY INITIATED  
Substance data SEARCH and crossover from CAS REGISTRY in progress...  
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

FULL SEARCH INITIATED 15:26:23 FILE 'REGISTRY'  
FULL SCREEN SEARCH COMPLETED - 262088 TO ITERATE

100.0% PROCESSED 262088 ITERATIONS 7979 ANSWERS  
SEARCH TIME: 00.00.01

L2 7979 SEA SSS FUL L1

L3 1042 L2

=> s l3 and py<2002  
21937596 PY<2002  
L4 708 L3 AND PY<2002

=> s l4 and (phenyl? or benzyl or oleyl)  
858171 PHENYL?  
189061 BENZYL  
9801 OLEYL  
L5 403 L4 AND (PHENYL? OR BENZYL OR OLEYL)

=> s l5 and ( tert-butylphenyl or dimethylbenzyl or oleyl)  
275795 TERT  
11468 BUTYLPHENYL  
9542 TERT-BUTYLPHENYL

10/923,271

(TERT(W)BUTYLPHENYL)

5702 DIMETHYLBENZYL  
9801 OLEYL

L6 1 L5 AND ( TERT-BUTYLPHENYL OR DIMETHYLBENZYL OR OLEYL)

=> d ibib abs hitstr

L6 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1949:17410 CAPLUS

DOCUMENT NUMBER: 43:17410

ORIGINAL REFERENCE NO.: 43:3360h-i,3361a-i,3362a-g

TITLE: Biosynthesis of penicillins. V. Substituted phenylacetic acid derivatives as penicillin precursors

AUTHOR(S): Corse, Joseph W.; Jones, Reuben G.; Soper, Quentin F.; Whitehead, Calvert W.; Behrens, Otto K.

SOURCE: Journal of the American Chemical Society (1948 ), 70, 2837-43

CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB cf. C.A. 43, 2274b. A description is given of substituted PhCH<sub>2</sub>CO<sub>2</sub>H derivs. which have been tested as precursor substances in the preparation of new penicillins. p-HOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CO<sub>2</sub>Et (I) (36 g.) and 38 g. PhCH<sub>2</sub>Cl in 300 mL absolute EtOH containing 13.3 g. MeONa, refluxed overnight and the ester refluxed

overnight with 70 g. KOH in 400 mL EtOH and 70 mL H<sub>2</sub>O, give 15.2 g. (p-benzyloxyphenyl)acetic acid (II), m. 120-1°. I (15.2 g.) in 200 mL H<sub>2</sub>O and 48.4 mL 4.135 N NaOH (stirred in an ice bath), treated dropwise with 12 g. ClCO<sub>2</sub>Et, the mixture stirred 2 h., and 32 mL 4 N HCl added, gives (p-carbethoxyoxyphenyl)acetic acid, m. 78-9°. II (15.2 g.) in 30 mL SOCl<sub>2</sub>, the mixture kept overnight, the residue treated with 11.7 g. DL-valine and 16 mL 12 N NaOH in 200 mL H<sub>2</sub>O, gives N-(p-benzyloxyphenylacetyl)-DL-valine (III), m. 144-5°, S 1.37 (S is the stimulation; compds. were tested at 0.0008 M concentration; the values represent the ratio units in test container/units in control container). The following analogs of III were prepared (R in RC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CONHCH(CHMe<sub>2</sub>)CO<sub>2</sub>H) (S is 1 unless otherwise given): o-NO<sub>2</sub> m. 173-5°, m-NO<sub>2</sub> m. 153-8° (S 0.88), p-NO<sub>2</sub> m. 134-5° (S 1.49), o-NH<sub>2</sub> m. 238-41° (S 1.37), p-NH<sub>2</sub> m. 220-7° (prepared by catalytic reduction of the NO<sub>2</sub> derivs.), o-Cl m. 122-4°, p-Cl m. 144-5° (S 1.33), p-CN m. 138-40° (S 1.24), p-I m. 148-50°, p-iso-Pr m. 114-15°, p-MeO m. 129° (S 1.52), 2,4,6-tri-Me m. 130-2° N-(p-nitrophenylacetyl)isoleucine m. 113-15°. The following esters were prepared by treating the substituted PhMe with Br and the resulting PhCH<sub>2</sub>Br with KCN, hydrolyzing the nitrile with aqueous alc. H<sub>2</sub>SO<sub>4</sub>, and esterifying with MeOH-H<sub>2</sub>SO<sub>4</sub>: Me (3,4-dibromophenyl)acetate m. 44-5° 3,4,5-tri-Br analog m. 78-9° 4-bromo-3-chloro analog m. 42-3°. Et (o-fluorophenyl)acetate, b24 135-6°, 52%; m-isomer, b28 126-9°, 22%; p-isomer, b31 128-30°, n25D 1.4776, 48%. Et (4-amino-3-nitrophenyl)acetate, bright yellow, m. 80-1° (68% on saturating the acid in EtOH with HCl and standing overnight). 3,4-MeO(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>Cl through the nitrile yields (4-methoxy-3-nitrophenyl)acetic acid, m. 122-5°. MeSPh (24.8 g.), 150 mL CS<sub>2</sub>, and 24 g. AcCl at 0°, treated with 30 g. AlCl<sub>3</sub> (in portions) and the mixture stirred 4 h., give p-methylmercaptoacetophenone (III), m. 72-5° 49.8 g. III, 9.6 g. S, and 27 mL morpholine, refluxed overnight, treated with 400 mL concentrated HCl and 300 mL H<sub>2</sub>O, and again refluxed overnight, give 25 g. (p-methylmercaptophenyl)acetic acid,

m. 92-4° Me ester b3 179-81°. m-F<sub>3</sub>CC<sub>6</sub>H<sub>4</sub>CN (51.5 g.) in 50 mL. ether, added (1 h.) to MeMgI (60 g. MeI) and, after 3 h., poured into 500 g. ice and 100 mL. concentrated HCl, gives 50% m-(trifluoromethyl)acetophenone (IV), b. 198-200°. m-F<sub>3</sub>CC<sub>6</sub>H<sub>4</sub>COCl (b750 184-6°, 95.5% yield) (93.5 g.) in 100 mL. ether, added dropwise to CdMe<sub>2</sub> (25 mg. Mg, 100 g. MeBr, and 110 g. CdCl<sub>2</sub>) in 700 mL. ether, gives 91% IV. IV (10 g.), 2 g. S, and 5.3 g. morpholine, heated 16 h. at 135°, treated with 30 mL. AcOH and 50 mL. concentrated HCl, and refluxed 7 h., give 89% [m-(trifluoromethyl)phenyl]acetic acid, m. 72-3°. p-PhOC<sub>6</sub>H<sub>4</sub>Ac (60 g.), 13 g. S, and 10 mL. morpholine, refluxed overnight, the crude product hydrolyzed (2 days) by refluxing with 75 g. KOH in 75 mL. H<sub>2</sub>O and 600 mL. EtOH, and the acid esterified with EtOH and H<sub>2</sub>SO<sub>4</sub>, give 25 g. Et (p-phenoxyphenyl)acetate, b0.2 173-4°. p-MeOC<sub>6</sub>H<sub>4</sub>CONHC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CO<sub>2</sub>H (m. 211-12°) and excess CH<sub>2</sub>N<sub>2</sub> in MeOH-ether give a quant. yield of the Me ester, m. 162°. Ph<sub>2</sub>S and AcCl give p-phenylmercaptoacetophenone, b1 180°, which, by the Willgerodt method and esterification, yields Et (p-phenylmercaptophenyl)acetate, b0.65 163°. I (36 g.) in 300 mL. absolute EtOH containing 11 g. MeONa, refluxed overnight with 30 g. Et<sub>2</sub>N(CH<sub>2</sub>)<sub>3</sub>Cl, gives 24 g. Et [p-(3-diethylaminopropoxy)phenyl]acetate, b0.3 145-7° (HCl salt, m. 121°).

p-HOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CONHCH<sub>2</sub>CH<sub>2</sub>OH (V) (49 g.) in 165 mL. 10% NaOH, treated with PhN<sub>2</sub>Cl (23 g. PhNH<sub>2</sub>) at 0°, gives 56.5 g. N-2-hydroxyethyl- $\alpha$ -(4-hydroxy-3-phenylazophenyl)acetamide, m. 180-1.5°. V (49 g.) and 79.7 g. Hg(OAc)<sub>2</sub> in 800 mL. 50% EtOH and 40 mL. AcOH, allowed to stand 12 days at room temperature and the solid product heated with 750 mL. 50% EtOH containing 5% AcOH, gives 51.4 g. N-2-hydroxyethyl- $\alpha$ -[3,5-bis(acetylmercuri)-4-hydroxyphenyl]acetamide, partially m. at 240° (rapid heating). p-tert-BuC<sub>6</sub>H<sub>4</sub>Ac (87 g.) through the acid (Willgerodt method), yields 19.4 g. Et (p-tert-butylphenyl)acetate, b0.47 95°. p-tert-AmC<sub>6</sub>H<sub>4</sub>Ac (68.5 g.) yields 15 g. Et (p-tert-amylphenyl)acetate, b2 124°. Reaction of I (90 g.) and 70 g. CH<sub>2</sub>:CHCH<sub>2</sub>Br, followed by esterification, gives 18.4 g. Et (p-allyloxyphenyl)acetate (VI), b0.5 126-7° oxidation of 44 g. VI in 100 mL. 70% Me<sub>2</sub>CO with 22 g. KMnO<sub>4</sub> in 300 mL. 70% Me<sub>2</sub>CO (with addition of 8 g. AcOH to the mixture) yields 24.8 g. Et [p-(2,3-dihydroxypropoxy)phenyl]acetate, b0.2 200°. N-2-Hydroxyethyl amides, RC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CONHCH<sub>2</sub>CH<sub>2</sub>OH, were prepared by heating the above and other esters with excess H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>OH overnight on the steam bath or several hrs. at 110-20° (R given; S is 1 unless otherwise given): p-acetamido m. 145-6°, p-allyloxy m. 84-5° (S 1.23), 4-amino-3-nitro m. 132°, p-NH<sub>2</sub> m. 103-4° (S 1.14), p-tert-Am oil, p-anisoylamino m. 210-11°, 4-bromo-3-chloro m. 104-6° (S 1.71), o-Br m. 106-7°, m-Br m. 129-30° (S 2.21), p-Br m. 108-9° (S 2.90), p-tert-Bu, oil, o-Cl m. 99-100°, m-Cl m. 114-17° (S 1.84), p-Cl m. 90-1° (S 1.97), 3,5-bis(acetylmercuri)-4-hydroxy, 3,5-dibromo-4-hydroxy m. 200-2°, 3,4-di-Br m. 125-7°, 2,4-di-Cl m. 118-19°, 3,4-di-Cl m. 113-14° (S 2.10), p-(3-diethylaminopropoxy) oil, p-(2,3-dihydroxypropoxy) oil (S 1.20), 3,5-diodo-4-hydroxy m. 179-80°, 2,3-di-MeO m. 93°, 3,4-di-MeO m. 96-8°, 3,4-di-Me m. 99-100° (S 1.27), p-EtO m. 90-1° (S 1.26), o-F m. 103-5° (S 1.23), m-F m. 75-7° (S 1.93), p-F m. 75° (S 1.54), o-HO oil (S 1.24), m-HO m. 92-3° (S 1.13), p-HO m. 110-12°, p-(2-hydroxyethylcarbamyl) m. 157-8°, 4-hydroxy-3-phenylazo m. 180-1.5°, m-I m. 127-9° (S 1.75), p-I m. 112-13° (S 1.83), 5-isopropyl-2-Me oil, p-iso-Pr oil (S 1.33), o-MeO oil, m-MeO m. 59°, p-MeO m. 86-8° (S 1.22), 3,4-methylenedioxy m. 99-100°,

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p-methylmercapto m. 115-17° (S 1.49), 4-methoxy-3-nitro m. 69°, o-Me m. 63-4° (S 1.36), m-Me oil (S 1.39), p-Me m. 76-8° (S 1.69), p-NO<sub>2</sub> m. 140-2°, p-PhO m. 95° (S 1.64), p-phenylmercapto m. 89-90°, p-Ph m. 172-5° (S 0.87), 3,4,5-tri-Br m. 212-13° (S 0.33), m-F<sub>3</sub>C oil (S 1.28), 2,4,6-tri-Me m. 144-5°. N-Allyl- $\alpha$ -(p-hydroxyphenyl)acetamide m. 84-6°. N-(2-Aminoethyl)- $\alpha$ -(p-methoxyphenyl)acetamide-HCl m. 135-8° (S 1.34). PhCH<sub>2</sub>CS<sub>2</sub>Me (18.2 g.) in 15 g. MePrNH on heating to boiling gives 86% N-methyl-N-propyl- $\alpha$ -phenylthioacetamide, b1.5 155-8°, n<sub>D</sub> 1.5876. The following phenylthioacetyl derivs. were prepared by exactly neutralizing the amino acid with 4 N NaOH, diluting with an equal volume of EtOH, adding 10% molar excess PhCH<sub>2</sub>CS<sub>2</sub>Me, and shaking for a few min. to several hrs.: D-penicillamine m. 132-3°, 55%; L-isomer m. 133-4°, 61%;  $\beta,\beta$ -diethoxyalanine, with 0.5 mol. H<sub>2</sub>O, m. 67.5-8°, 84%; DL-valine m. 102-3°, 95%; DL-isoleucine m. 95-6°, 75%. Details are given of the formation of p-HOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CONHCH<sub>2</sub>CH<sub>2</sub>OH. From the results of the S data it is difficult to draw any generalizations. Both the kind and position of the substituents had a marked influence upon the ability of the compound to act as a penicillin precursor. That the nature of the PhCH<sub>2</sub>CO<sub>2</sub>H derivative had a profound influence upon its utilization by the mold was illustrated in several cases.

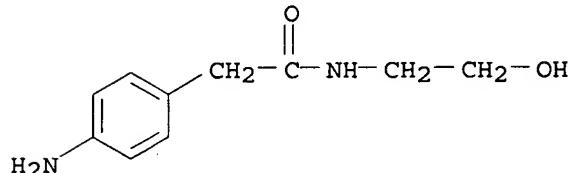
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855925-26-7P, Acetamide, 2-(4-amino-3-nitrophenyl)-N-2-hydroxyethyl-

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(preparation of)

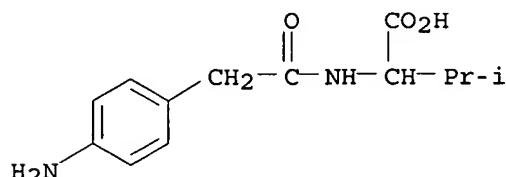
RN 167613-92-5 CAPLUS

CN Benzeneacetamide, 4-amino-N-(2-hydroxyethyl)- (CA INDEX NAME)



RN 855701-79-0 CAPLUS

CN Phenaceturic acid, p-amino- $\alpha$ -isopropyl- (5CI) (CA INDEX NAME)



RN 855925-26-7 CAPLUS

CN Acetamide, 2-(4-amino-3-nitrophenyl)-N-2-hydroxyethyl- (5CI) (CA INDEX NAME)

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